FATIGUE STRENGTH OF GLASS: A CONTROLLED FLAW STUDY

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The fatigue strength properties of glass containing Vickers indentation flaws are described. The responses are found to be highly sensitive to the state of the flaws, notably to the presence or otherwise of irreversible contact stresses or of deformation-induced radial cracks. When radial cracks are present (post threshold state) the data can be described completely in terms of conventional fracture mechanics laws. Removal of the residual stresses (by annealing) results in higher strengths and reduced fatigue susceptibility. When radial cracks are not present (subthreshold state), as is the case at

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sufficiently small contact loads, the data deviate from the extrapolated predictions of macroscopic crack theory. The observed strengths are higher than equivalent post threshold levels, with increased fatigue susceptibility and greater scatter. It is concluded that the sharp-crack concept of flaws remains valid down to the threshold load for crack initiation, but that below this threshold it is the crack precursor processes which control the failure properties. Implications of these results concerning the mechanical response of optical fibers are considered.

I. INTRODUCTION

In previous papers in this volume[1,2] it was argued that indentations could be used to simulate natural flaws in glass surfaces. The convenience of predetermining the geometry and location of strength-controlling flaws, so that the mechanics of failure could be investigated by direct inspection during actual testing, was proposed as a unique advantage of this approach. In particular, the role of residual contact stresses (responsible for creating the flaws in the first place), and the changing nature of the flaws on traversing the threshold load for radial crack generation, were emphasized as critical elements in the characterization of strength properties. Correlations between the failure characteristics of post threshold indentation flaws and machining, abrasion and scratching damage were used to establish the general applicability of fracture mechanics concepts to surfaces with typical handling damage.

In this paper we make use of the indentation method to determine the time-dependent strength, i.e. "fatigue," properties of glasses. First we investigate the post threshold region, with definitive tests to demonstrate the important role of residual stresses in the evaluation of crack velocity parameters. Macroscopic crack laws are shown to hold for all flaw sizes within this region. Then the subthreshold region is studied. Significant deviations from extrapolated post threshold responses are found, indicative of a transition from propagation-controlled to initiation-controlled fracture processes.

II. DYNAMIC FATIGUE ANALYSIS: POST THRESHOLD REGION

The fracture mechanics approach for analyzing the fatigue of glass in reactive environments is based on the underlying assumption that fatigue failure occurs by subcritical extension of a dominant microscopic crack to an instability configuration. Provided the rate of crack extension is defined by a unique function of the crack driving force, the analysis of fatigue strength follows from two basic starting equations: one equation represents the crack velocity function, $v = dc/dt = v(K)$, where c is a characteristic crack dimension, t is time and K is the stress intensity factor; the other represents

the crack driving force, $K = K(\sigma_a, c)$, where $\sigma_a = \sigma_a(t)$ characterizes the applied loading. Combination of these two equations yields a differential equation in crack length and time which can be solved for arbitrary service conditions once the parameters of the two equations are known. Then, incorporation of the instability condition $K = K_c$, $dK/dc > 0$, where K_c is the toughness, allows failure stresses σ_f to be evaluated.

In this section we confine our attention to the response of indentation flaws formed at loads in excess of the threshold, i.e. at $P > P_c$.[1] The stress intensity factor for this configuration is given by[2]

$$K = \chi P/c_r^{3/2} + \psi\sigma_a c_r^{1/2} \tag{1}$$

where c_r is the characteristic crack dimension, with χ and ψ representative parameters of the residual field and crack geometry, respectively. The crack velocity function most widely used is[3]

$$v = v_o(K/K_c)^n \tag{2}$$

where n and v_o are kinetic constants for a given material-environment system. Experimentally, it is convenient to load the crack system under "dynamic fatigue" conditions, i.e. $\sigma_a = \dot{\sigma}_a t$ with $\dot{\sigma}_a = $ const. Inserting this loading specification into Eqs. (1) and (2) then produces our differential equation, the solution of which has the general power law form[4-6]

$$\sigma_f P^{1/3} = \left[\lambda_p' \dot{\sigma}_a P\right]^{1/(n'+1)} \tag{3}$$

where n' and λ_p' are load-independent parameters. We may note that, for a given flaw size (as determined by P), the failure stress σ_f should plot linearly with stressing rate $\dot{\sigma}_a$ on a logarithmic dynamic fatigue plot, in exactly the same way as for conventional fatigue theories based on simplistic "Griffith" (residual-stress free) flaws.

There are two features of the solution in Eq. (3) which bear some elaboration here. The first of these concerns the way the slope and intercept terms, i.e. n' and λ_p', relate to the crack velocity parameters, n and v_o.[5] For a crack without residual stresses the slope term identifies directly with the velocity exponent, i.e.

$$n' = n \qquad (\chi = 0) . \tag{4a}$$

The corresponding connecting expression for cracks with unrelaxed residual stresses is

$$n' = 3n/4 + 1/2 \qquad (\chi \neq 0) . \tag{4b}$$

In the event that part of the residual stress field is relieved prior to strength testing (to the point that the flaw size condition $c_o' < c_m$ alluded to in Ref. 2 is violated), some relationship intermediate between Eqs. (4a) and (4b) will apply. Analogous (albeit somewhat more complicated) connecting expressions are available for the λ_p' terms.[5,6]

The second feature of interest in Eq. (3) is the explicit appearance of the indentation load. Thus, for a given material-environment system, the effect of varying P should be manifested as a simple translation of the data line on a fatigue plot. This, as we shall see, provides us with a convenient means for systematically investigating the influence of flaw size on the fracture mechanics relations.

III. RESULTS: INFLUENCE OF RESIDUAL STRESS

Let us now survey the experimental evidence in support of the formulation for post threshold flaws given in Sect. II, with particular reference to the role of residual contact stresses. We first review the results of controlled indentation studies, and then examine how well the analysis can be extended to a configuration with natural, viz. abrasion, flaws.

A. INDENTATION FLAWS

The strongest indication of residual stress influences in the strength properties of glasses has come from comparative tests on indented surfaces in their immediate and annealed states. Mention has already been made of such tests, in connection with inert strengths, in Ref. 2. There it was shown that the annealing effectively removes all residual driving forces on the indentation cracks, leading to an increase in strength. We need now to review the corresponding effects on fatigue characteristics.

Dynamic fatigue tests have been run on Vickers-indented soda-lime glass in water.[4] The results for as-indented and annealed surfaces are shown in Fig. 1. It is immediately clear that the slopes of the two curves, as well as their heights (cf. inert strength cutoff levels), are significantly different, as expected from Eqs. (3) and (4). Thus for the annealed surfaces we obtain $n' = 17.9 \pm 0.5$ from the sloped (fatigue) portion of the curve, which corresponds, within experimental error, to macroscopically determined values of the crack velocity exponent n (e.g. using double cantilever beam[3]). For the as-indented surfaces we similarly have $n' = 13.7 \pm 0.2$; this transforms, via Eq. (4b), to $n = 17.6 \pm 0.3$, consistent with the preceding evaluation. Similar consistency is found for the crack velocity coefficient v_o, as determined from the intercept term λ_p'.[4] The solid curves shown in Fig. 1 are actual re-evaluations of the entire fatigue responses from the starting differential equation, using these parameter evaluations.
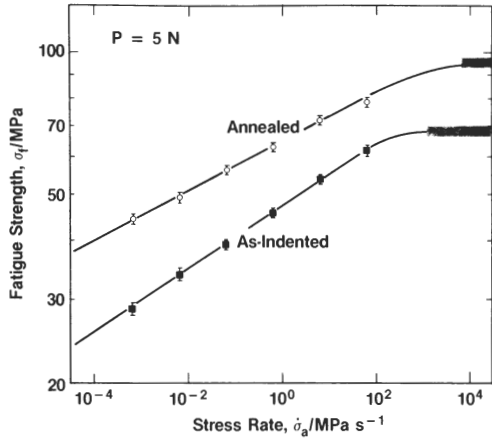
Fig. 1. Dynamic fatigue of Vickers-indented soda-lime glass, in water. Data at single contact load, for as-indented and post-indentation annealed specimens. Error bars are standard deviation limits, 10-30 tests per point. Shaded bands indicate inert strength levels. After Ref. 4.

An additional check on the theoretical formulation was obtained in the original study[4] by directly monitoring the entire subcritical crack evolution to failure for both flaw types represented in Fig. 2, at one specified stress rate. The results are shown in Fig. 2. The solid curves again are predictions from the master differential equation, using the parameters determined from Fig. 1. It is seen that the as-indented flaw proceeds to failure more quickly than its
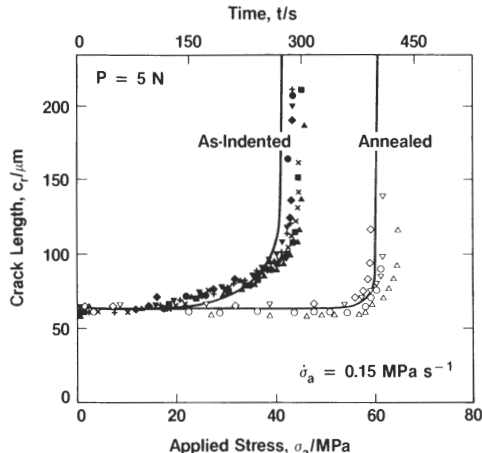

Fig. 2. Response of indentation cracks in soda-lime glass, in water, to applied stress. Data at fixed stressing rate, for as-indented and post-indentation annealed specimens. After Ref. 4.

annealed counterpart, once more illustrating the strong influence of the residual driving term.

The results of this study have important implications in the analysis of fatigue behavior. The most obvious of these comes straight from Fig. 2, in which the approach to failure instability clearly involves a stage of extensive subcritical crack growth. Another comes from the fact that the two curves in Fig. 1, although different in their positions on the plot, are of essentially the same form, viz. a linear fatigue portion with inert strength cutoff. Thus, without *a priori* knowledge of the state of the flaw it would be impossible to tell solely from fatigue data on any given surface to what extent, if any, residual stresses influence the fracture mechanics.

B. ABRASION FLAWS

Some results for grit-abraded soda-lime glass[7] illustrate how the principles outlined above for ideal indentation flaws may be applied to naturally damaged surfaces. Fig. 3 shows the dynamic fatigue response for such surfaces tested in water. A curve fit to the linear sloped portion of the fatigue curve in accordance with Eq. (3) yields $n' = 15.7 \pm 0.7$. This value lies intermediate between those obtained from the two extreme cases in Fig. 1, implying that some residual stress relief about individual abrasion contact sites must have occurred. Direct observation of the damage sites revealed chipping, suggesting that the relief mechanism is one of extensive lateral crack growth.[2] The solid curve in Fig. 3 is once more generated from the fatigue differential equation, using the indentation-determined crack
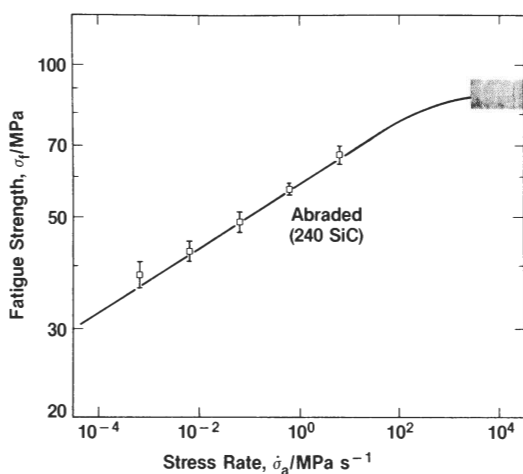

Fig. 3. Dynamic fatigue of abraded soda-lime glass, in water. Error bars are standard deviation limits, for 10-30 tests per point. After Ref. 7.

velocity parameters from Sect. II but with residual-stress and crack-geometry terms χ and ψ calibrated from inert strengths.[7] The degree of agreement between predicted curve and experimental data in this plot may be taken as a measure of the applicability of the fracture mechanics concept to handling damage of this kind.

IV. RESULTS: INFLUENCE OF FLAW SIZE

The indentation flaws investigated in the previous section were of a size (see Fig. 2) at the upper extreme of natural handling damage in glass. Such flaws are seen from routine optical observations to behave in the same way as macroscopic cracks. The question now arises as to how far we may extrapolate the fracture mechanics theory of Sect. II into the realm of microscopic flaws. In particular, what response might we expect as we enter the subthreshold region? Systematic studies by Dabbs et al.,[8,9] using indentation load P as a primary test variable, provide some answers to these questions, and are accordingly summarized below.

A. POST THRESHOLD FLAWS

We first analyze data from the post threshold region, again for soda-lime glass in water.[8] The load range covered was from $P = 10N$ down to 0.5N, corresponding to a flaw size (characteristic dimension of hardness impression) range from 30 μm to 2 μm. At the lower end of this range the radial cracks did not form spontaneously in laboratory atmosphere; instead, they were "chemically induced" by immersion in a dilute solution of HF.[8] This conveniently provided a means by which fatigue data for both post threshold and subthreshold flaws could be obtained at overlapping load ranges.

The data for the post threshold flaws are plotted in Fig. 4. The lines represent appropriate least-squares fits, obtained by regressing over all the data in accordance with Eq. (3). Thus the effect of varying flaw size is simply to shift the position of the curve, without change of slope, on the fatigue plot. The fact that the degree of fit holds equally well for "small" as for "large" flaws is a strong indication that the sharp-crack base of fracture mechanics theory has general applicability in this region.

B. SUBTHRESHOLD FLAWS

Let us now examine the corresponding results for the subthreshold flaws.[9] In this case the data are somewhat more limited in range, owing to the increasing tendency for failure to occur at spurious handling flaws as the indentation load is lowered. Nevertheless, with careful surface preparation prior to testing the subthreshold flaws do provide favored sites for fracture, and all data presented here are of this failure type.
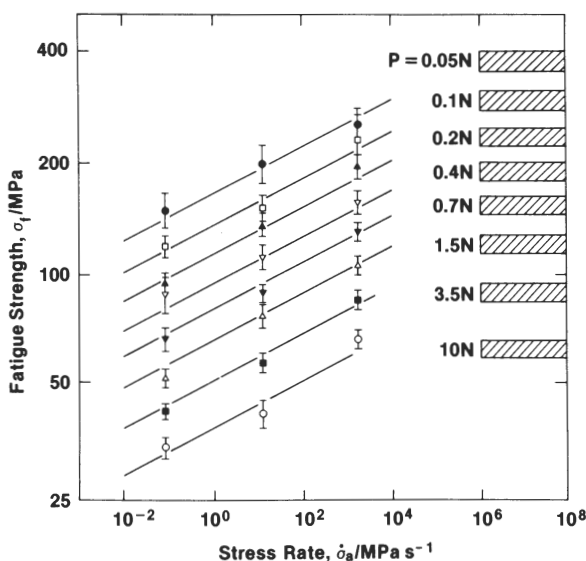
Fig. 4. Dynamic fatigue of Vickers-indented soda-lime glass, in water. Data at several contact loads. Error bars are standard deviation limits, minimum of 6 tests per point. Shaded bands indicate inert strengths. After Refs. 6 and 8.

Unfortunately, unlike the post threshold region, we have no detailed theoretical description upon which to devise a fatigue plotting scheme for subthreshold flaws. The best we can do at present is to retain the use of Eq. (3), realizing that this equation must now be regarded as nothing more than an empirical formula for manipulation. Accordingly, Fig. 5 represents the usual fatigue plot, but with P specifically incorporated into the axes to facilitate data reduction onto universal fatigue curves. Inclusion of the post threshold data (from Fig. 4) is simply to emphasize that the subthreshold flaws have undergone some distinctive changes in their strength-controlling characteristics. Thus it is clear that flaws created at a specified contact load are significantly less severe if radial cracks have not popped in. On the other hand, the subthreshold flaws are more susceptible to fatigue. From analysis of the slopes we have $n' = 9.0 \pm 0.8$ for these flaws, which compares with $n' = 14.0 \pm 0.3$ for their post threshold counterparts. Recalling that $n = 17.9 \pm 0.5$ for annealed flaws (Sect. III), this represents an ultimate reduction of about a factor of two in the apparent crack velocity exponent. The scatter is also wider for the subthreshold flaws, suggestive of a more stochastic process of failure.

These results are consistent with a transition in the fracture mechanism at failure, from propagation-controlled to initiation-controlled instabilities. In
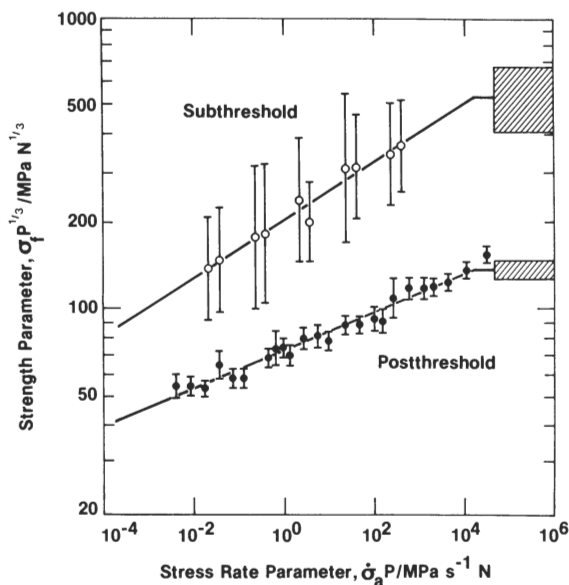
Fig. 5. Dynamic fatigue of Vickers-indented soda-lime glass, in water. Data at various indentation loads, reduced to universal curves by appropriate choice of coordinate variables. Error bars for subthreshold data are standard deviations, minimum of 20 tests per point. Shaded bars are inert strengths. After Ref. 9.

this context it may be noted that the inert strength for post threshold flaws lies below the fatigue strength for subthreshold flaws over a large portion of the stressing rate range, so that, when radial cracks do initiate during a fatigue test, failure must occur spontaneously from the precursor deformation-induced nuclei within the confines of the hardness impression.[1]

V. DISCUSSION

The results described in Sects. III and IV clearly show that the nature of flaws is an important consideration in fatigue characterization. A flaw of given size may respond in a number of different ways, depending on whether there are residual stresses present or whether cracks have been formed. This is particularly important in the context of lifetime design. Thus, on removing residual stresses from flaws in glass the lifetimes can be increased by $\approx$ 3 orders of magnitude, Fig. 1; similarly, by suppressing crack initiation corresponding increase of $\approx$ 6 orders of magnitude can be realized, Fig. 5. Lifetime design procedures should therefore always be based, as far as possible, on the actual surface conditions which are to operate in service, without extrapolations beyond the data range. This philosophy is, of course, commonly preached by many in the glass reliability fraternity, particularly

those who seek to characterize strength properties in terms of statistical distribution functions. Our approach does more than merely reinforce this design philosophy, however; it provides physical insight into the failure mechanisms themselves, e.g. shear fault decohesion,[1] and thereby offers the prospect for combating these mechanisms in a scientific manner.

One corollary from the above discussion is the potential role of flaw history in the fatigue behavior. It could be argued that aging of newly created flaws should be beneficial, since residual stresses will tend to relax with time (especially in reactive environments, Ref. 2), gradually shifting the response from the lower to the upper curve in Fig. 1. However, this benefit would only be felt if radial cracks were present at all stages. If the flaws begin their life in the subthreshold state, as they are more likely to do with components in the ultra-high strength region, the danger exists that radial cracks may initiate spontaneously at some subsequent stage of service. This danger may increase dramatically under certain operating conditions, e.g. hostile chemical and thermal environments, in which case premature failures may occur. There is some evidence from the optical fiber literature[11,12] that small-scale natural flaws can indeed suffer abrupt increases in severity with prolonged exposures to aqueous environment. Under such conditions no amount of control strength testing (including proof testing) would be capable of predicting the lifetimes of components.

Our tests here have been confined to one glass, soda-lime, so we have to be careful in drawing strong conclusions about optical fibers, which are usually manufactured from fused silica. We have already indicated that soda-lime and fused silica glasses are respectively "normal" and "anomalous" in their indentation responses,[1] in which case the general applicability of the effects described in Sects. III and IV has to be demonstrated. This calls for controlled flaw studies on actual fibers themselves. An earlier, preliminary study of this kind[13] did indeed confirm that fibers undergo the same abrupt increase in strength on reducing the indentation load below the threshold point. More recent experiments on the fatigue behavior of subthreshold-indented fibers[14] have shown that the fatigue susceptibility also follows the same trends as those reported here for soda-lime glass, namely a diminished value of the apparent crack velocity exponent relative to that for the postthreshold region, to about one half of the true exponent. These results could help to explain the widespread "discrepancies" in literature n values noted elsewhere in this volume.[15]

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DISCUSSION

Q: R. D. Maurer. The "subthreshold" flaw data could very well be relevant to optical fibers. However, the extrinsic flaws there come from a wide variety of sources, not only abrasion. In those particular cases where abrasion flaws dominate, behavior of this type might be experimentally confirmed.

A: B. R. Lawn. The subthreshold phenomenon described here, although specifically addressed in terms of indentation flaws, is expected to be a general phenomenon; i.e. there is a well defined size effect above which cracks are formed (propagation-controlled) and below which they are not (initiation-controlled). In the latter case it is the precursor processes actually responsible for generating the cracks in the first place which enter the kinetic description. It seems to me that the flaws in optical fibers are more likely to be of this type, since extrapolations of macroscopic crack laws into the region of ultra-high strengths tend to be in conflict with experimental data on fibers. Further work in this area is clearly needed.

Q: P. K. Gupta. 1. You show that for subthreshold flaws the "apparent" n value is lower than that for post-threshold flaws. Ritter mentioned this morning that n for pristine glass samples is higher than that for abraded samples. Does it mean that subthreshold indentation flaws do not simulate flaws in pristine high stress glass samples?

2. For subthreshold flaws, your data show a reasonably good fit to the predictions of crack propagation theory (except that n value is different). It appears to me that just because n value is different does not justify rejecting crack propagation ideas for subthreshold flaws.

A: B. R. Lawn. 1. It seems to me that n for pristine fibers is subject to considerable variation: some report it higher than the corresponding value for abraded surfaces, some lower. In our experiments we obtain values which are typically one half the macroscopic values, and this appears to be reasonably consistent with the relatively low n found with fresh fibers. The truth of the matter is that we simply do not know what the nature of ultra-small flaws is, and we should therefore be cautious when identifying these flaws with any simplistic picture, e.g. the "sharp crack" hypothesis implicit in most slow crack growth models.

2. I do not agree that our subthreshold flaws show "a reasonably good fit" to the predictions of crack propagation theory. We have demonstrated that extrapolations of data from the macroscopic domain are not valid in the microscopic domain. However, this does not exclude crack growth models either. It is possible that slow crack growth does occur in the subthreshold region, but the residual stress field through which this growth takes place is more complex than our existing models can handle. On the other hand, it is also possible that the kinetics of crack initiation might be controlled by the rate dependence of the precursor shear fault process, in which case the slow crack growth ideal is lost. So far, definitive evidence one way or the other has not been forthcoming, although our results do appear to lean toward the second of the two possible explanations.